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## COMPLETE SPECIFICATION

### An improved Process for the Preparation of Higher Aliphatic Alcohols.

We, SOCIETE BELGE DE L'AZOTE ET DES PRODUITS CHIMIQUES DU MARLY, a Belgian Body Corporate, of Liege, Belgium, do hereby declare the invention, for which we 5 pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an improved process for the preparation of higher aliphatic alcohols (known as heavy alcohols or fatty alcohols), and is concerned with such a process in which the starting materials are fatty materials including animal and vegetable 15 fats and oils.

It is known that fatty alcohols can be prepared by direct catalytic hydrogenation of fatty materials. However, this method 20 degrades the glycerol which is contained in the fatty materials in the form of glycerides of higher fatty acids, and which is reduced by the direct hydrogenation reaction into the commercially unimportant isopropyl alcohol and propylene glycol. Furthermore this 25 direct hydrogenation method leads to an elevated consumption of catalysts and hydrogen.

Alternatively, a preliminary hydrolysis of the fatty materials to recover the glycerol 30 frees fatty acids which considerably complicate the direct catalytic hydrogenation owing to their strong corrosive action or which must be transformed into esters (e.g., methyl esters) before their hydrogenation. 35 It was therefore considered desirable to prepare the latter esters directly from the fatty materials by transesterification or alcoholysis of these materials and to convert the fatty esters into fatty alcohols by catalytic 40 hydrogenation under pressure. It was also sometimes desirable to transform the unsaturated fatty alcohols formed by the treatment of raw materials including unsaturated fatty acids into saturated fatty alcohols. 45 According to the present invention there

is provided an improved continuous process for the preparation of higher fatty alcohols from fatty materials which include vegetable and animal oils and fats, wherein the fatty materials are first catalytically treated under 50 superatmospheric pressure and at elevated temperatures by a monohydroxy aliphatic alcohol, the alcohol being in a proportion in excess of the stoichiometric amount whereby glycerine separates from the fatty materials 55 which are transformed into esters of said alcohol and of the carboxylic acids combined in said materials, the excess alcohol and the glycerol being thereupon separated from said esters, and wherein the esters are 60 catalytically transformed by means of hydrogen in excess under superatmospheric pressures and at elevated temperatures into higher fatty alcohols, the excess hydrogen and the freed monohydroxy alcohol being 65 separated from said fatty alcohols, and the latter alcohol being recycled to the transesterification stage.

A new apparatus for the transesterification and hydrogenation reactions is also 70 provided.

If methanol is used as the monovalent alcohol, the alcoholysis is effected at a pressure of at least 100 kg/cm<sup>2</sup> and at a temperature of about 250°C. by passing the reaction mixture over a zinc silicate catalyst 75 of particular composition. The products resulting from this operation, i.e., the glycerol, the methyl esters of the fatty acids and the excess methanol reactant are separated by expansion, distillation, washing and decantation steps, the glycerol product being sufficiently concentrated and pure for commercial use and the recovered excess 80 methanol being rectified and recycled.

As regards the methyl esters which are formed, these may be directly conducted into the hydrogenation units where they are reacted with hydrogen at a pressure of at least 300 kg/cm<sup>2</sup> and at fixed temperatures over 85

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one or more specific hydrogenation catalysts.

If the initial fatty materials include glycerides of unsaturated fatty acids and it is desired to produce only saturated alcohols, the hydrogenation must be effected in the presence of two different types of catalysts operating successively at different temperatures instead of in the presence of a single type of catalyst.

The first of these two types of catalyst is, for instance, a mixture of zinc and copper chromite and is used at a temperature of between 300°C. and 360°C. for the reduction of the esterified fatty acids into alcohols whilst the second is of the nickel silicate type and is used at a temperature of between 180°C. and 250°C. for the saturation of the double linkages of the unsaturated alcohols which are still present in the reaction mixture resulting from the primary hydrogenation.

In the case in which the whole or part of the unsaturated fatty alcohols obtained by the primary hydrogenation are to be recovered, either the secondary hydrogenation is omitted or only part of the reaction products leaving the primary hydrogenation chamber may be subjected to the secondary hydrogenation in such manner that the iodine index is adjusted as desired.

Owing to the fact that the two kinds of catalyst function at characteristically different temperatures, i.e., for instance, at about 325°C. for the first catalyst type and at about 200°C. for the second type, the reaction mixture must be partially cooled between the two hydrogenation stages. By contrast, it is not necessary or even advisable to separate between the two stages the methanol which is liberated by the primary hydrogenation since the methanol does not hinder the secondary hydrogenation stage and can be recovered without any additional difficulties and recycled to the alcoholysis stage during the separation of the fatty alcohol products.

For a better understanding of the invention and to show how the same is to be carried into effect, reference will now be made to the accompanying drawings in which:—

Fig. 1 is a flow sheet of one constructional form of an apparatus for preparing higher fatty alcohols from fatty materials including animal and vegetable fats and oils; and

Fig. 2 shows diagrammatically an enlarged detail of the flow sheet of Fig. 1.

Referring firstly to Fig. 1, the oil or fat to be treated which may be made or maintained fluid by preheating and which is fed through a conduit 1, and methanol which is fed to the conduit 1 by a conduit 2, are mixed in a pump 3. The methanol is fed in a proportion in excess of that required for

the alcoholysis reaction.

The mixture of the alcohol and the fat or oil which is compressed to a pressure of about 100 kg/cm<sup>2</sup> and elevated to a temperature of about 250°C. by compression and preheating units (not shown in Fig. 1) is introduced into the lower end of a catalyst tube 4 which includes a zinc silicate catalyst and is fully insulated. The transesterification reaction is effected in this catalyst tube at a temperature in the neighbourhood of 250°C.

The reaction mixture which leaves the catalyst tube 4 comprising methyl esters of fatty acids, glycerol and excess methanol is exhausted through a conduit 5 to an intermediate point in a column 6 and is rapidly expanded therein from a pressure of 100 kg/cm<sup>2</sup> to a pressure of between 1.2 and 2 kg/cm<sup>2</sup> in order to distil off the major part of the methanol from the methyl esters and the glycerol, whilst the glycerine-containing waters from a washing column 14 are led into the top of the column 6 by a conduit 15. The methanol vapours are conducted by a conduit 7 into a rectification column 8 from which the methanol which is recovered by cooling and resulting condensation is recycled by a conduit 9 to the pump 3.

The mixture of methyl esters, glycerol, residual methanol and water is conducted by a conduit 10 to a decantation tank 11 at the bottom of which crude glycerol is abstracted through a conduit 12 at a concentration of 80%-92%. The supernatant liquid is led through a conduit 13 into a washing column 14 in which the remaining glycerol is separated from the esters by washing with water. The washing water is fed, as indicated above, to the column 6 by the conduit 15 in order to facilitate the subsequent decantation of the glycerol in the decantation tank 11.

The methyl esters which have been washed and separated from the glycerol-containing waters by decantation in the column 14 are fed by a conduit 16 to a reservoir 17. A compressor 18 compresses the esters to a pressure of 400 kg/cm<sup>2</sup> and supplies them to a mixer 19 in which they are admixed with hydrogen at a pressure of 400 kg/cm<sup>2</sup> which is supplied by a conduit 20.

The intimated mixture of methyl esters and hydrogen under a pressure of 400 kg/cm<sup>2</sup> is firstly preheated in a heat-exchanger 21 by means of part or all of the products from the hydrogenation treatment and is then elevated to a temperature of 325°C. in a preheater 21a. The preheated mixture is then introduced into a reaction chamber 22 of a first hydrogenation unit (or tube) in which a copper and zinc chromite catalyst causes the methyl esters of the fatty acids to be transformed by an exothermic reaction into fatty alcohols and methanol. Owing to the elevated temperature of the

catalytic reaction, the thin-walled reaction chamber 22 which is constructed of stainless steel is itself enclosed in a power tube which is not directly exposed to the elevated temperatures and has carbon steel walls of sufficient thickness to withstand the reaction pressures. This mode of construction whilst considerably reducing the cost of the catalyst tubes also ensures great safety in operation. By means of baffles contained within the reaction chamber 22 and between which the catalyst is maintained, the segregation of the mixture of esters and hydrogen and the occurrence of preferential channels which the hydrogen might form in the catalytic mass are avoided. A perfectly homogeneous mixture reacting under stable thermal conditions without deactivation or lack of use of certain parts of the catalyst is maintained.

The hydrogenation on a zinc and copper chromite catalyst in the catalyst tube 22 is principally directed to the carboxylic ester groups which is reduced to the hydroxylic group, and only partially affects the double linkages of the unsaturated fatty acids. Except in the case in which the original fatty materials only include saturated fatty acids, the mixture leaving the tube 22 will thus contain both unsaturated and saturated fatty alcohols together with methanol and excess hydrogen, so that the fatty alcohol products might have a high iodine index showing an unsaturation which is undesirable for certain uses of these alcohols.

The unsaturated alcohols may be transformed into the corresponding saturated alcohols by conducting the reaction mixture leaving the tube 22 by means of a conduit 23 firstly into a cooling system 24 in order to lower the temperature from 325°C. to about 190°C. and then into the reaction chamber 25 of a second hydrogenation unit (or tube) having a construction analogous to that of the first hydrogenation unit (or tube), but containing a nickel silicate catalyst and operating at an average temperature of about 180°C.-200°C.

By means of automatic means for heat regulation (not shown in the diagram) which act on the preheating units 21 and 21a and on the cooling unit 24, the reaction mixture is fed into the entrance of each of the two catalytic tubes at a temperature in the neighbourhood of that in which the appropriate reaction is initiated, i.e., to a temperature of about 335°C. on entering the first catalyst tube and 190°C. on entering the second tube. The exothermic hydrogenation reactions thus take place in the most favourable manner, i.e., under thermally stable conditions which are easily controllable by regulation of the hydrogen feed and which only lead to a negligible formation of secondary products which can easily be eliminated sub-

sequently from the mixture of higher fatty alcohols and methanol obtained after the removal of the excess hydrogen.

As indicated above, the hydrogenation reaction products partially preheat the reaction mixture to be hydrogenated. Thus, the products are fed through a conduit 26 to the heat-exchanger 21 in which they are cooled to 80°C. and from which they are led by a conduit 27 to a separator 28 from which excess hydrogen is recycled always under pressure by a conduit 29 to the mixer 19. At the same time, the mixture of fatty alcohols and methanol is led by means of a conduit 30 firstly to an expansion unit 31 in which the pressure of the mixture is reduced in two stages from 400 kg/cm<sup>2</sup> firstly to 25 kg/cm<sup>2</sup> and then to 2 kg/cm<sup>2</sup> and is then led to a distillation unit 32 for the separation of methanol which is recycled by conduits 33, 9 and 2 to the pump 3, and of fatty alcohols which are conducted by a conduit 34 to the final purging and supply units.

It will be understood that various modifications of the individual units described above and of their manner of operation may be effected. Thus, as indicated in Fig. 2, the two-stage hydrogenation may be effected in the same vessel in which the reaction mixture leaving the preheater 21a passes successively over a zinc and copper chromite catalyst in a compartment 22', then through a cooling coil 24', and finally over a nickel silicate catalyst in a compartment 25' before being exhausted through a conduit 26.

Furthermore, the transesterification reaction itself may be effected in two stages, the glycerol which is formed being withdrawn at an intermediate stage in order to improve the glycerol yield. By slight modifications, the apparatus described allows also the recovery, in admixture with the saturated alcohols which they accompany and from which they may be separated by known means, of unsaturated alcohols obtained by the transesterification of unsaturated fatty materials, and the subsequent hydrogenation on a zinc and copper chromite catalyst only of the alcoholysis products. For this purpose, the reaction products from the hydrogenation tube 22 are partly or wholly abstracted from the conduit 23 by a conduit 35 and led through the heat exchanger 21 to the separation and purification units.

In order that the thermal balance for the process as a whole should be improved additional regenerators or heat-exchangers may be employed in order for instance, to utilise to an increased extent the heat evolved by the two hydrogenation stages for heating the various distillation and purification units.

The catalysts to be used in the various stages of the process may be prepared in the following manner:—

1. *The powdered zinc silicate catalyst for the transesterification.*

A solution of zinc acetate is prepared by introducing 70 Kg of 85% acetic acid into a suspension of 40 Kg of zinc oxide in 100 litres of water which is heated to 70°C. The zinc acetate solution is poured with vigorous mixing into a solution of 200 litres of commercial 36° Be sodium silicate in 2,000 litres of water. The resulting mixture is diluted to 3,000 litres and the precipitate which is formed is separated by decantation and washed with water until the washing water is free of salts. The washed precipitate is successively centrifuged and extruded, and then heated firstly at a temperature of between 40°C. and 50°C. until the precipitate is sufficiently consistent and finally at 130°C. Between 100 and 110 Kg of catalyst are thus obtained.

2. *Copper and zinc chromite catalyst for the hydrogenation of the carboxylic ester group.*

14 Kg of zinc oxide and 125 Kg of chromic oxide are successively added to a solution containing 10 Kg of cupric oxide, 180 Kg of 85% acetic acid and 500 litres of water. The mixture is heated until the solid constituents are entirely dissolved, and 500 litres of powdered active carbon previously dried at about 130°C. are then poured into the boiling solution. 500 litres of zinc and copper chromite catalyst are then obtained by evaporating the solution and by agitating the mixture during the evaporation.

3. *Nickel silicate catalyst for the saturation of the olefinic linkages.*

A commercial sodium silicate solution of 36% concentration (having approximately the composition  $\text{Na}_2\text{O} \cdot 3\text{SiO}_2$ ) is treated with a 20% caustic soda solution in order to obtain a product having the composition  $\text{Na}_2\text{OSiO}_3 + 1/10 \text{Na}_2\text{O}$ . A solution of nickel nitrate in 250 litres of water containing 20 kgs. of metallic nickel and partially neutralised to a pH of about 6.5 by the addition of powdered sodium carbonate is then poured into the alkaline sodium silicate solution. The pH is adjusted to about 7 by the addition of the nickel solution and the resulting mixture is diluted with water to a volume of 3,000 litres. The nickel silicate precipitate is separated by decantation and is washed until the washings do not contain any salts. Fibrous asbestos previously washed in 10% nitric acid is malaseated into the nickel silicate precipitate in a proportion of 1% (5% of the dry mass to be obtained). The precipitate is first tamped and progressively dried at 40°C.-50°C. to a sufficiently solid consistency, and finally

dried at 130°C. About 50 kgs. of catalyst are obtained.

The invention is illustrated by the following examples in which the methanol obtained in the hydrogenation stage is recycled for use in the transesterification stage:—

EXAMPLE 1. A mixture of 1,270 kgs. of coconut oil and 533 kgs. of methanol compressed to 100 atmospheres was introduced at a volumetric space rate of 0.5 litres of oil per hour per litre of catalyst into a transesterification tube heated to a temperature of 250°C. and having a zinc silicate catalyst. The reaction products leaving the catalyst tube were rapidly expanded in order that the major part of the excess methanol should be removed, and the methyl esters were separated by decantation from 142 kgs. of glycerine (in 85% solution). The esters were then hydrogenated by means of a large excess of hydrogen at between 320°C. and 325°C. under a pressure of 400 atmospheres, and the crude fatty alcohols were purified by rapid expansion from the methanol which is freed by the hydrogenation reaction, and were then distilled to yield 1000 kgs. of distilled alcohols. Since the transesterification reaction leads to an equilibrium, the yield of recovered glycerine in a single pass does not easily exceed 85%. If the transesterification reaction is effected in two phases, and the glycerine is separated between the two phases, a glycerine recovery of 90% is easily exceeded. For this purpose, the mixture of methyl esters, untransformed coconut oil and glycerine together with excess methanol was re-cooled in a heat-exchanger and was conducted under pressure at approximately 50°C. into the decanter. The glycerine which contains water and methanol was continuously withdrawn whilst the mixture of esters and residual coconut oil together with additional methanol (to replace methanol entrained by the glycerine) was again subjected to the reaction in a second transesterification tube as indicated in Example 2 below.

EXAMPLE 2. A mixture of 633 kgs. of methanol and 1,270 kgs. of coconut oil was compressed to 100 atmospheres, and fed to a first transesterification tube at a temperature of 250°C. to 280°C. and having therein a zinc silicate catalyst. 125 kgs. of water were injected into the mixture of residual coconut oil, methyl esters, glycerine and excess methanol which had been cooled to 15°C. A quantity of methanol equal to that which had been withdrawn with the glycerine was added to the decanted esters and the mixture which was firstly reheated to 260°C. was led into a second reaction tube, at a volumetric space rate of 1 litre of mixture per hour per litre of catalyst, in order to complete the transesterification reaction. The major part of the excess methanol was

removed by rapid expansion and 151 kgs. of 85% glycerine solution were separated from the reaction products by decantation. The methyl esters compressed in the presence of 5 a large excess of hydrogen to 400 atmospheres were hydrogenated at a temperature of between 325°C. and 330°C. over a copper and zinc chromite catalyst yielding after a rapid expansion and distillation 1,000 kgs. 10 of fatty alcohols.

In the above examples relating to the treatment of coconut oil, the hydrogenation was limited to the reduction of the carboxylic ester groups of fatty acids by copper and 15 zinc chromite catalysts which do not affect the greater part of the double linkages in unsaturated fatty acids. The following example illustrates a two-stage hydrogenation.

20 EXAMPLE 3. A mixture of 1,220 kgs. of tallow grease having an iodine index of 55, and 458 kgs. of methanol was preheated to 250°C. and compressed to 100 atmospheres. The hot compressed mixture treated as in 25 Example 1 yielded 115 kgs. of glycerine in 85% solution.

The methyl esters in admixture with a large excess of hydrogen were compressed to 400 atmospheres and preheated at 325°C., 30 the heated and compressed mixture being passed into a first hydrogenation tube having a copper and zinc chromite catalyst. The mixture of alcohol products whose iodine index was between 30 and 35 was recooled to 200°C. and passed into a second hydrogenation tube having a nickel silicate catalyst. 35 The reaction products were rapidly expanded in order to remove the major part of the methanol, and yielded on distillation 1,090 kgs. of crude alcohols having an iodine index of between 0.3 and 0.5. 40

An analogous treatment in two hydrogenation stages of coconut oil was effected, according to the following example.

45 EXAMPLE 4. In an apparatus such as that shown in Fig. 1, 1,270 kgs. of coconut oil and 633 kgs. of methanol were introduced into the transesterification tube 4, the mixture being passed through the tube at a volumetric space rate of 0.5 litres per hour of oil per litre of catalyst under a pressure of 100 50 kg/cm<sup>2</sup> and at a temperature of 250°C. over a zinc silicate catalyst. By conducting the reaction products into the column 6 and by rapidly expanding them thereinto a pressure of 1.2 kg/cm<sup>2</sup>, 482 kgs. of methanol were recovered by volatilisation followed by condensation and were recycled. 55

142 kgs. of glycerol in 85% solution were 60 withdrawn from the decantation tank 11 and the ester mixture from the transesterification reaction was purified from residual glycerol by washing with water in the column 14. The wash water from this purifying operation was conducted into the column 65

6 so that the glycerol still remaining in the esters after decantation could be recovered in the tank 11 and separated in that tank from the methyl esters.

The methyl esters which had been successively subjected to hydrogenation firstly on a copper and zinc chromite catalyst under a pressure of 400 kg/cm<sup>2</sup> and at a temperature of 325°C. in the catalyst tube 22, and then over a nickel silicate catalyst at a pressure of 400 kg/cm<sup>2</sup>, and at a temperature of 200°C. in the catalyst tube 25 yielded after expansion and separation of the reaction product mixture into its constituents 1,060 kgs. of crude fatty alcohols including mainly 80 C8 to C18 alcohols together with 100 kgs. of recycled methanol. The iodine index of the coconut oil employed was 10 and that of the fatty alcohol products was 0.3.

What we claim is:—

85 1. An improved continuous process for the preparation of higher fatty alcohols from fatty materials which include vegetable and animal oils and fats, wherein the fatty materials are first catalytically treated under 90 superatmospheric pressure and at elevated temperatures by a monohydroxy aliphatic alcohol, the alcohol being in a proportion in excess of the stoichiometric amount, whereby glycerine separates from the fatty materials which are transformed into esters of said alcohol and the carboxylic acids combined in said materials, the excess alcohol and the glycerol being thereupon separated from said esters, and wherein the esters are 100 catalytically transformed by means of hydrogen in excess under superatmospheric pressures and at elevated temperatures into higher fatty alcohols, the excess hydrogen and the freed monohydroxy alcohol being 105 separated from said fatty alcohols, and the latter alcohol being recycled to the transesterification stage.

2. A process as claimed in Claim 1, wherein the transformation of the fatty materials into said esters of said monohydroxy alcohol is carried out in the presence of a zinc silicate catalyst. 110

3. A process as claimed in Claim 1 or 2, wherein said monohydroxy alcohol is 115 methanol.

4. A process as claimed in any of Claims 1 to 3, wherein the transesterification is effected at a pressure of at least 100 kg/cm<sup>2</sup>, and at a temperature of between 250°C. and 120 280°C.

5. A process as claimed in any of the preceding claims, wherein the major part of the excess monohydroxy alcohol from the transesterification reaction is recovered by 125 rapid expansion of the reaction mixture and recondensation of the vaporised monohydroxy alcohol.

6. A process as claimed in any of the preceding claims wherein the glycerol is separated 130

rated from the esters of said monohydroxy alcohol by washing with water, decantation and concentration of the aqueous glycerine extract by evaporation.

5 7. A process as claimed in any of the preceding claims, wherein the esters of said monohydroxy alcohol are hydrogenated and thereby transformed into a mixture of saturated and un-saturated higher fatty alcohols  
10 in the presence of a mixed zinc and copper chromite catalyst.

8. A process as claimed in Claim 7, wherein the reaction in the presence of the zinc and copper chromite is carried out at  
15 a pressure of 400 kg/cm<sup>2</sup> and at a temperature of between 320°C. and 330°C.

9. A process as claimed in Claim 7 or 8 wherein the unsaturated alcohols in said mixture are saturated by catalytic hydrogenation in the presence of a nickel silicate  
20 catalyst.

10. A process as claimed in Claim 9, wherein the saturation of the fatty alcohol mixture is effected at a temperature of between 180°C. and 200°C. and at a pressure  
25 of 400 kg/cm<sup>2</sup>.

11. A process as claimed in Claim 10, wherein the two reduction operations are effected separately, the mixture from the  
30 first hydrogenation in the presence of the copper and zinc chromite being cooled to about 200°C. before being conducted over the nickel silicate catalyst.

12. A process as claimed in Claim 11, wherein the two hydrogenation operations are effected in two separate chambers, the reaction mixture leaving the copper and zinc chromite catalyst in one compartment being cooled to about 200°C. in an intermediate  
40 compartment before being conducted over the nickel silicate catalyst.

13. A process as claimed in any of the preceding claims, wherein the excess hydrogen separated from the hydrogenation reaction mixture is recycled, a major part of said  
45 monohydroxy alcohol being thereupon separated from the hydrogenation reaction mixture by a rapid expansion of said mixture, and wherein the remainder of the freed

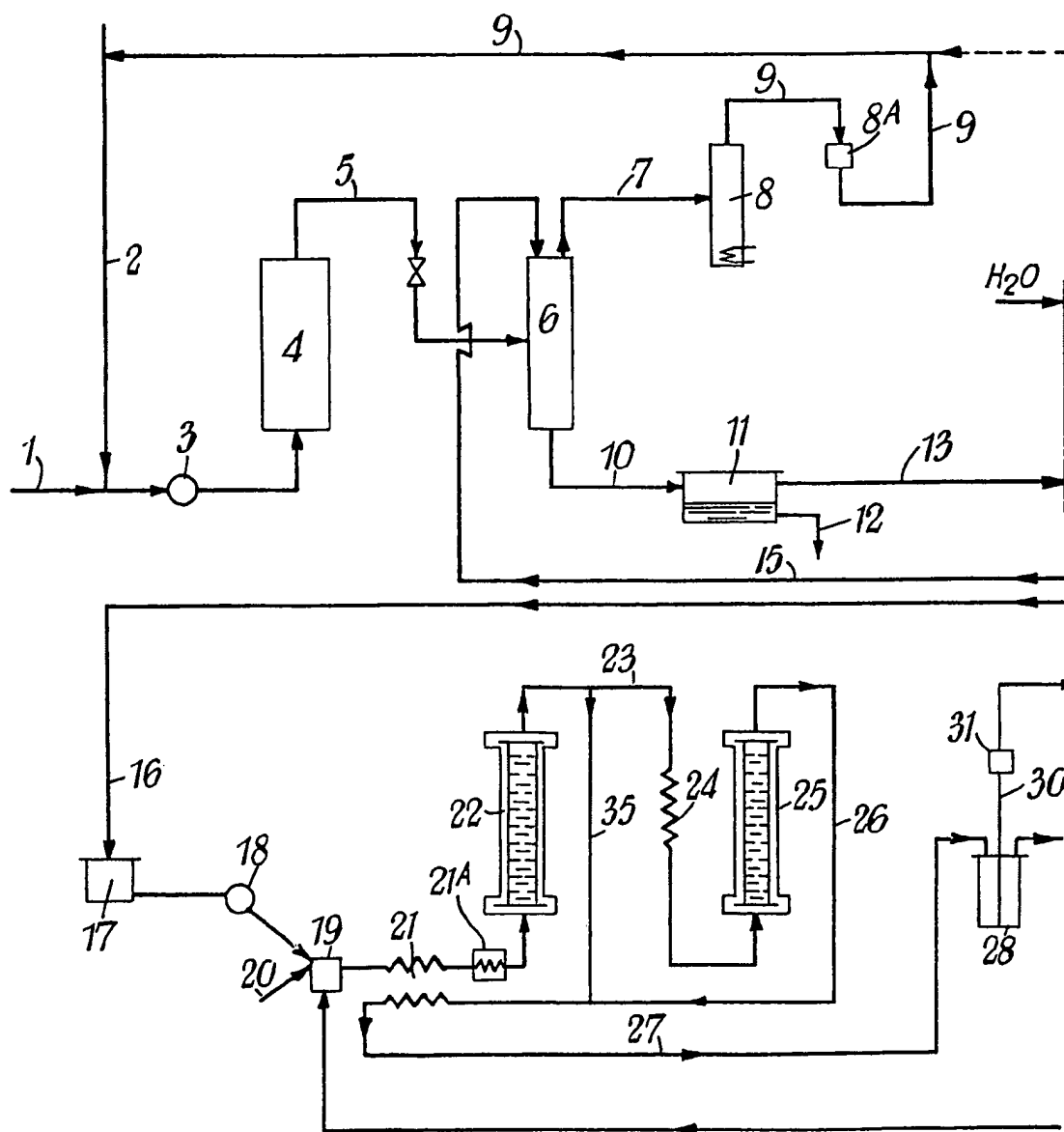
alcohol is separated from the higher fatty 50 alcohol product by distillation.

14. A process for the preparation of higher fatty alcohols from fatty materials comprising vegetable and animal fats and oils, wherein the fatty materials are first 55 treated by methanol in excess over a zinc silicate catalyst under a pressure of about 100 kg/cm<sup>2</sup> and at a temperature of between 250°C. and 280°C., the major part of the excess methanol being recovered by rapid 60 expansion and recondensation, wherein glycerol is separated from the resulting esters of methanol by washing with water, decantation and concentration of the aqueous extract by evaporation, wherein the ester mixture is transformed into saturated fatty alcohols in two stages in the presence of excess hydrogen at a pressure of 400 kg/cm<sup>2</sup>, the ester mixture being first conducted over a zinc and copper chromite catalyst at a temperature of between 320°C. and 330°C. and then over a nickel silicate catalyst at a temperature of between 180°C. and 200°C., the excess hydrogen being separated from the hydrogenation reaction mixture and re- 75 cycled, and wherein a major portion of the freed methanol is separated from the higher hydrogenation reaction mixture and recycled fatty alcohol products by rapid expansion followed by recondensation, the remainder 80 of the freed methanol being separated by distillation and the total freed methanol being recycled to the transesterification stage.

15. An improved process for the preparation of higher fatty alcohols from fatty 85 materials including vegetable and animal fats and oils, substantially as hereinbefore described with reference to any one of the specific examples, and the appendant drawing.

16. Glycerol and higher fatty alcohols 90 whenever prepared by the process claimed in any one of Claims 1 to 15.

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*FIG. 1.*

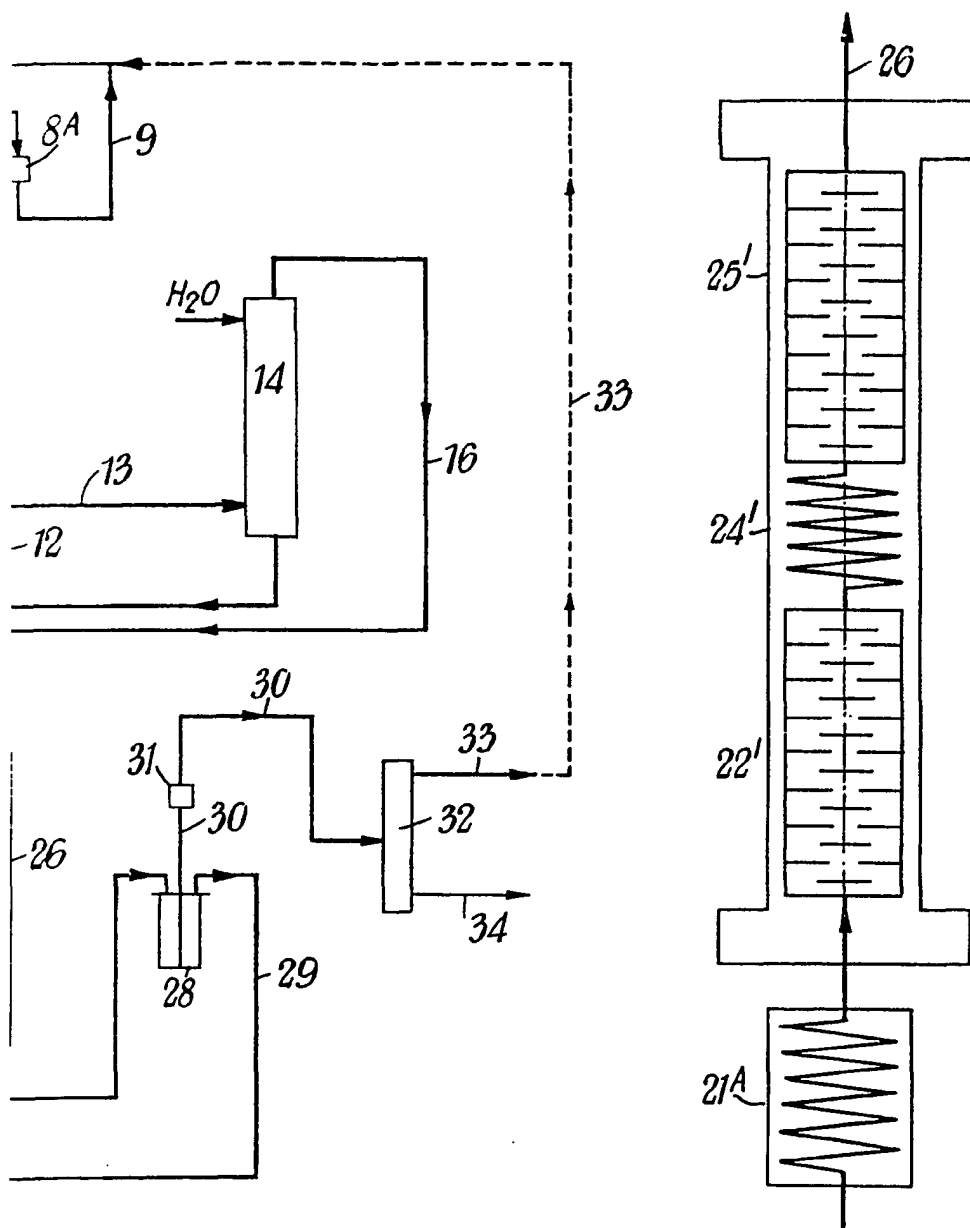


FIG. 2.



